38 VOLATILE TRACE CONGENERS IN ALCOHOLIC BEVERAGES BY GC/FID

38.1 Summary

38.1.1   The identification and quantitation of the trace congeners is necessary to establish the identity and quality of distilled alcoholic spirits. Liquid samples are directly injected into a gas chromatograph equipped with a flame ionization detector. Using an external primary standard, the concentrations of the congeners are determined and used to help identify and characterize distilled alcoholic spirits.

38.2 Specimen Requirements

38.2.1   Approximately 3 mL liquid or 3 g solid material

38.3 Reagents and Standards

38.3.1   Ethanol, 95% (grain alcohol or equivalent)

38.3.2   Acetaldehyde

38.3.3   Methanol

38.3.4   Acetone

38.3.5   2-propanol

38.3.6   Methyl acetate

38.3.7   1-propanol

38.3.8   Diacetyl

38.3.9   Ethyl acetate

38.3.10 2-butanol

38.3.11 Isobutyl alcohol

38.3.12 1-butanol

38.3.13 Acetal

38.3.14 Acetoin

38.3.15 3-Methyl-1-butanol

38.3.16 2-Methyl-1-butanol

38.3.17 Ethyl lactate

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38.4   Solutions, calibrators and controls

38.4.1   Stable Congener Standard. To a 1 L volumetric flask containing approximately 900 mL 95% ethanol, add the following volumes (or weights) of congener standard to obtain the final concentrations. Standard is stable for 2 years.

Congener	mL of standard	Final Concentration (g/100L)
1-butanol	0.3	24.30
1-propanol	0.2	16.08
2-butanol	0.2	16.20
2-methyl-1-butanol	0.5	40.80
2-propanol	0.2	15.60
3-methyl-1-butanol	1.5	121.95
acetoin	0.05 grams	5.0
acetone	0.2	15.80
isobutyl alcohol	0.5	40.10
methanol	0.5	39.60

38.4.2   Primary Congener Standard. Add approximately 9.5 mL of stable congener standard to a 10 mL volumetric flask. Add the following volumes of labile congeners and QS to volume with stable congener standard. Stable for 3-5 days.

Congener	μL of standard	Final Concentration (g/100L)
acetaldehyde	2	15.66
methyl acetate	2	18.66
ethyl acetate	7	63.07
diacetyl	2	19.62
acetyl	2	16.40
ethyl lactate	2	20.60

38.4.3   Quality control beverages

38.4.3.1   Vodka (negative control)

38.4.3.2   Bourbon whiskey quality control sample

38.4.3.3   Illegally distilled quality control sample

38.5   Apparatus

38.5.1   Agilent GC/FID, Chemstation software, compatible computer and printer

38.5.2   1.8 mL GC autosampler vials

38.5.3   GC/FID parameters. Conditions may be changed to permit improved performance.

38.5.3.1   Column: Restek RTX 1301, 60 m x 0.25 mm

38.5.3.2   Direct injection: 2 μL sample

38.5.3.3   Oven program

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Equilibration Time	0.5 min
Initial Temp	50º C
Initial Time	10 min
Ramp 1	5º C/min
Final Temp 1	130º C
Ramp 2	40º C
Final Temp 2	210º C
Injector	150º C
Detector (FID)	200º C
Hydrogen flow	30 mL/min
Air flow	400 mL/min
Combined flow	25 mL/min
Make-up gas	helium
Inlet	Split
Split ratio	50:1
Split flow	143 mL/min
Total flow	148 mL/min
Pressure	41 psi

38.6   Procedure

38.6.1   Transfer approximately 1.5 mL standard, controls and case samples to GC autosampler vials.

38.6.2   Analyze case samples in duplicate.

38.6.3   Run vodka samples (negative control) throughout the sequence to ensure no carryover of congeners.

38.6.4   Inject 2 µL on GC/FID.

38.7   Calculation

38.7.1   Congeners are identified based on relative retention times compared to primary standard. Identification is performed by instrument software. Retention times for analytes must be within $\pm 2\%$ of the retention time obtained from the primary standard.

38.7.2   Concentration is calculated automatically by the software based on a single point calibration curve of the primary standard. Congener concentration is based on comparison of peak height (area) between case sample and primary standard.

38.8   Quality Control and Reporting

38.8.1   Positive controls. Acceptable tolerance for congener controls (Bourbon whiskey and illegally distilled quality control) is $\pm 20\%$ of the target concentration (previously established mean for that control).

38.8.2   Negative control. The negative control (vodka) is used to check for carryover. An acceptable negative control may not contain congeners other than ethanol.

38.8.3   Duplicate tolerance. Calculate the average of the case sample duplicates. Calculate 5% of the average and a $\pm 5\%$ range. Replicates must be within the $\pm 5\%$ range. Reanalyze the sample if it is outside of tolerance.

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38.8.4   By comparison of congener distribution and congener ratios between the unknown sample and previously analyzed distilled spirits, a beverage class can often be determined.

38.8.5   Congener values are never reported. The values are solely used to determine the class, brand or nature of the alcoholic beverage.

38.9   References

38.9.1   Amerine, M. *Laboratory Procedures for Enologists*. UC Davis, 1967.

38.9.2   Barnett, J.H. and J.R. Einsman. *Occurrence and Distribution of Congeners in Distilled Alcohol Spirits*. J Assoc of Official Analytical Chemists Vol 60, 1977.

38.9.3   Lange, N.A. *Lange's Handbook of Chemistry*. New York: McGraw-Hill, 1967.

38.9.4   Lembeck, H. *Grossman's Guide to Wine, Beers and Spirits*. New York: Charles Scribner's Sons, 1983.

38.9.5   Lichine, A. *Alexis Lichine's Encyclopedia of Wines and Spirits*. New York: Alfred Knopf, Inc., 1983.

38.9.6   *Official Methods of Analysis of the Association of Official Analytical Chemists*. 15th Ed., 1990.